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Original Article

An *in-vitro* evaluation of mechanical and esthetic properties of orthodontic sealants

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ABSTRACT

Objective: To evaluate mechanical and esthetic Properties of two commercially available orthodontic sealants: Opal®Seal (OS) and L.E.D. Pro Seal (PS). **Materials and Methods:** Discs of each sealant were prepared to test the following properties: Micro hardness, wear resistance and color stability. Samples were randomly selected after the wear test for SEM imaging to analyze surface morphology. **Results:** OS was significantly harder than PS ($P < 0.001$). PS was significantly more wear resistant than OS ($P < 0.05$). PS showed a greater ΔE*ab (increased staining) when placed in wine or coffee showing a significant difference ($P < 0.05$). SEM showed particle size, shape and distribution were different for PS and OS reflecting the pattern seen on wear surfaces. **Conclusion:** Both orthodontic sealants are beneficial for protecting enamel. However with better wear properties PS was superior in resisting mechanical stresses. OS was more color stable.

Key words: Esthetics, micro hardness, orthodontic sealants, wear properties, white spot lesions

INTRODUCTION

The prevalence of white spot lesions (WSL) in orthodontic patients is an ever growing problem. Patients receiving orthodontic treatment are at significantly higher risk of developing WSL than untreated patients.[1-6] Incidence of WSL during orthodontic treatment was 73-95%.[5,6] Orthodontists encounter this problem despite the oral hygiene instructions delivered routinely in addition to the recommendations for fluoride treatment and professional cleaning. WSL on tooth surfaces is not only an esthetic consideration, but also a risk factor for developing cavitated lesions. This necessitates the need for noncompliance approach to protecting the enamel surfaces during orthodontic treatment.

By mechanically sealing enamel surface, teeth can be protected from acid-induced demineralization. Materials that could be used as sealants on tooth surfaces require characteristics including resistant to oral environmental stresses such as brushing, food, saliva, and resistant to color change for esthetic reasons.

Routine oral hygiene practices of orthodontic patients are not adequate to prevent the occurrence of WSL. In patients with WSL the risk of developing further lesions continued to be elevated for the next 5 years.[3]

Oral hygiene instruction has been shown to have a minimal influence on patients undergoing orthodontic treatment as evidenced by a short-term reduction in plaque for up to 5 months.[7]

Fluoride containing toothpastes and casein phosphopeptide-amorphous calcium phosphate

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(CPP-ACP) complex (MI Paste™ and MI Paste Plus™, GC America, Alsip, IL) were shown to protect enamel surfaces from dental caries.[8,9] However, application of CPP-ACP did not produce a significant reduction in WSL.[10] CPP-ACP application requires patient cooperation as trays with paste needs to be placed in the mouth daily for 3-5 min.[9] The difference in reduction of WSL in patients who had CPP-ACP application in the above studies could be due to dissimilarity of patient's compliance. Most of the fluoride treatment protocols rely heavily on patient's compliance. In the light of these results, compliance -free preventive systems or treatment agents would be of interest to clinicians and patients.

Two currently available photo-cure surface sealers with Glass Ionomer particles and nano-fillers used for bonding brackets to etched enamel surfaces are Opal®Seal (OS) (Ultradent, Salt Lake City, UT) and L.E.D Pro Seal (PS) (Reliance Orthodontic Products, Itasca, IL, US). The application of sealers provides a mechanical barrier to the acid and prevents the demineralization.

The aim of this study was to evaluate the physical properties of these two commercially available sealants in terms of surface hardness, wear resistance and color stability.

MATERIALS AND METHODS

Wear resistance

A total of 10 wear resistant test specimens (15 mm diameter × 2 mm thick discs) were prepared from two commercially available orthodontic sealers, (OS, L.E.D PS). Liquid sealers were applied to the mold and covered with a glass microscopic slide. Specimens were photo-polymerized through the glass slide using a photo curing unit (Demetron Optilux 400, Kerr, Orange, CA, US) for 30 s with a light intensity of 250 mW/cm^2 checked with a curing radiometer (Demetron Model 100, Kerr). After polymerization, the sample surfaces were polished with 600-grit, 800-grit and 1200-grit SiC papers to remove matrix rich surface layer and to standardize the surface texture and flatness. Samples were stored in deionized water for 24 h at 37°C before testing. Wear resistance of each material was evaluated using a pin on disk testing apparatus (SPI-Tribotester Model 500, Spire, Bedford, MA, US). The pin consists of 1/16 inch diameter nylon rod positioned perpendicularly to the sealant specimen surface. The nylon pin was rotated across the sealant surface to produce a circular wear track approximately 10 mm in diameter. The specimens were abraded with 50 g pin load for 180 min, at 85 rpm, in toothpaste slurry prepared by mixing 25 g of toothpaste (Procter and Gamble, Cincinnati, OH, US) in 25 mL of deionized water. The toothpaste slurry was renewed after each experimental test cycle was completed. A contact profilometer (Surftest SJ-400, Mitutoyo, Kawasaki, Japan) was used to measure the cross section wear track profile at eight different sites. Software (MountainsMap®, Digital Surf, Besançon, France) was used to calculate surface area of the wear track cross section. The outside and inside dimensions of the wear track were measured using a traveling microscope (MM-11, Nikon, Tokyo, Japan). The volume of sealant material abraded was estimated by finding the product of the mean values of the eight wear track surface area measurements and the circumference of wear track. The values of each group were compared using standard *t*-test ($P < 0.05$).

Micro hardness test

A total of 10 discs of each sealant was prepared as described previously for the hardness test. Discs were stored in distilled water for 24 h at 37°C. After 24 h, the surface hardness of the specimen was measured using a hardness tester (MS-1, Shimadzu, Kyoto, Japan) under 25 g load for 15 s with the long axis of the diamond indenter parallel to the specimen's surface. Three measurements were recorded for each of the 10 specimens. The values obtained were converted to Knoop hardness numbers (KHNs). Differences between test group KHN values was done using a two-tailed *t*-test with the significance level set to $P < 0.05$.

Colorimetric test

A total of 15 specimens of each orthodontic sealer were prepared using a round plastic mold (25 mm diameter and 2 mm thickness) for the colorimetric test. Immediately after polymerization, baseline color values of all samples were measured using a spectrophotometer (Minolta CM-2002, Konica-Minolta Ewing, NJ) with the specular component excluded geometry under D65 illumination over a white background. Thereafter, 5 specimens of each sealant were placed in one of three solutions, distilled water, coffee or red wine. The time points for colorimetric data taken using the above-mentioned device were 24 h, 3 days, 1 week and 2 weeks. As each specimen was tested, they were run under distilled water for 5 s and patted dry with blotting paper to confirm their

surfaces were dry but not desiccated. The color differences (ΔE*ab) between baseline and after aging were calculated by CIE Lab color-difference formula: $\Delta E^*ab = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]$. Two-way analysis of variance (ANOVA) was used to compare the color changes between baseline and after 2 weeks for two materials, followed by the Newman–Keuls multiple comparisons test. For a comparison between the changes of the color over time (i.e. 24 h, 3 days, 1 week and 2 weeks) in each material, repeated measures ANOVA, followed by the Newman–Keuls multiple comparisons test was used ($P < 0.05$).

Scanning electron microscopy

Three samples were randomly selected from each surface sealant group after wear resistance test for scanning electron microscopy (SEM) imaging. Images were recorded at the wear track surfaces and also away from wear track surfaces as controls. Quanta 200 FEG environmental SEM was used for imaging.

RESULTS

Pro Seal was found to have a significantly greater wear resistance compared to OS ($P < 0.05$) [Figure 1]. Examples of wear tracks of each sealant traced by a contact profilometer were shown in Figure 2 demonstrating the surface area and volume affected. OS, showed an average volume of wear of 0.031 mm^3, when compared to PS's 0.013 mm^3, showing significant difference ($P < 0.01$).

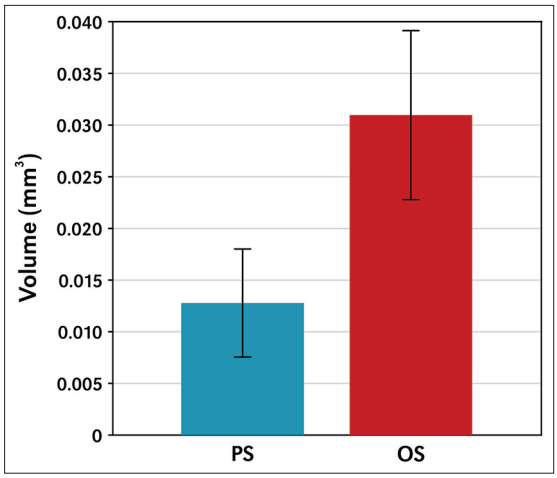

Figure 1: Average wear volume loss (mm^3) of Pro Seal (PS) and Opal seal (OS) as measured by a pin on disk testing apparatus with toothpaste slurry. OS shows greater volume loss with significant difference with PS ($P < 0.05$)

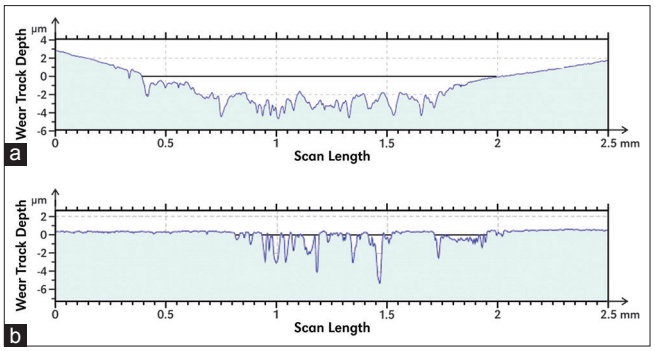

Figure 2: Wear tracks of Opal seal (OS) (a) and Pro Seal (PS) (b) traced by a contact profilometer. Average volume of wear of OS is significantly greater than that of PS ($P < 0.01$)

Micro hardness of the samples was measured in KHN [Figure 3]. OS demonstrated greater hardness with a significant difference ($P < 0.001$).

Overall, significant color changes occurred when the samples were placed in wine [Figures 4 and 5]. The OS shows better color stability when compared to PS. For both sealants, wine had the highest ability to stain when compared to water and coffee, as demonstrated by higher ΔE*ab values ($P < 0.05$). When comparing the two sealants to each other at 2 weeks, PS exhibited significantly greater Δ E*ab when stored in wine and coffee ($P < 0.05$). Both sealers showed changes over time that were significant when stored in water ($P < 0.05$) for 2 weeks and showed percievable color changes (OS: ΔE*ab 7.21, L.E.D. PS: ΔE*ab 8.81). When OS was placed in the wine, there was an initial rapid change in ΔE*ab followed by minimal changes occurred after the initial color changes. When PS was placed in the wine, a continuous increase in color occurred over time showing significant changes at each time interval ($P < 0.05$). When OS was compared to itself at 2 weeks in different mediums, no significant differences were shown, however when PS was compared with itself at 2 weeks, a significant difference is shown when stored in wine ($P < 0.05$).

The SEM images in Figure 6c show that the particle in the OS composites was larger than 250 nm. Figure 6a and c shows that OS contains both large and small sizes of polygonal filler particles. PS when viewed under the lower magnification the particle size was too small to be resolved. In high-resolution imaging, PS particle of spherical shape was measured <100 nm [Figure 6d]. These particles clustered closely together, rather than distributed uniformly.

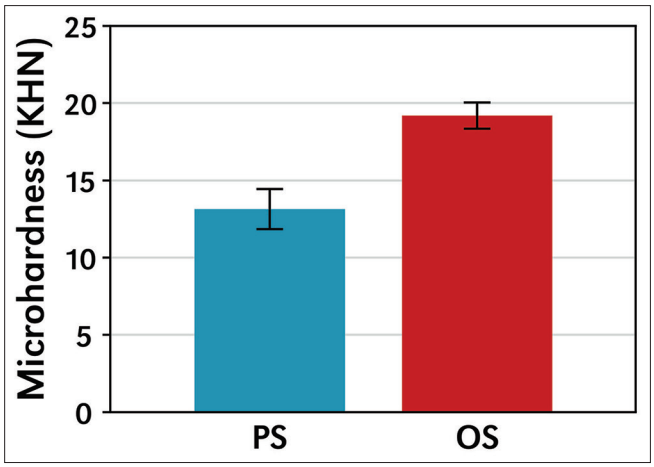

Figure 3: Average micro hardness (Knoop hardness number) of Pro Seal (PS) and Opal seal (OS). Surface hardness of the OS is significantly greater than that of PS ($P < 0.001$)

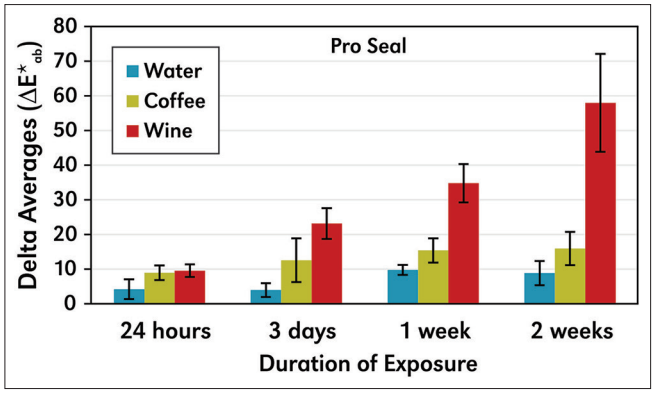

Figure 5: Color changes (ΔE*ab) of Pro Seal in three different media. Colorimetric evaluation was performed by a spectrophotometer

DISCUSSION

This study measured the wear resistance, micro hardness and color stability of PS with 18% filler content and OS with 38% filler content, to evaluate the suitability of these products as an orthodontic sealant. Since PS and OS are available as enamel sealants in the market and used in orthodontic practices, we investigated the physical properties and esthetic properties, which could be of clinical relevance for an orthodontic sealant. In this regard, materials that could provide mechanical sealing of enamel surfaces for a longer period would be of tremendous interest to clinicians.

Pro Seal and OS are filled with glass ionomer and nano filler particles of varying amounts.

PS exhibited better wear resistance and significantly higher protection to demineralization than a fluoride varnish and a conventional unfilled sealant in a laboratory environment.[11,12] There was no remarkable

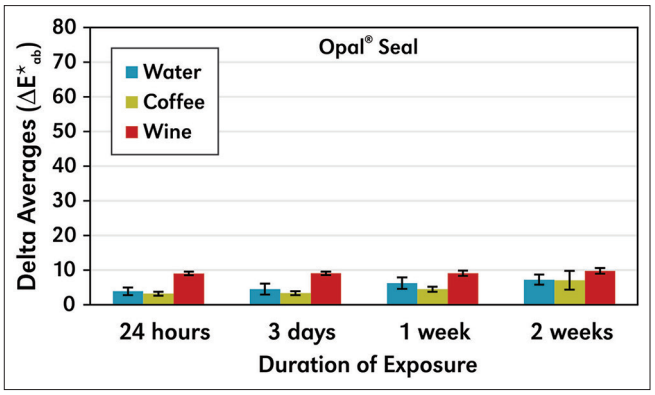

Figure 4: Color changes (ΔE*ab) of Opal seal in three different media. Colorimetric evaluation was performed by a spectrophotometer

difference in PS thickness as a result of wear caused by brushing when compared with an un-brushed side of the same tooth.[13]

Pro Seal includes a propriety catalyst which allows complete polymerization without oxygen inhibited layer and therefore leaves no porosity and resists tooth brush abrasion and normal wear for up to 2 years.[14] Majority of the resin sealants employ camphorquinone as a photoinitiator with the maximum light absorber.[15] Sealants with camphorquinone as a catalyst will leave an oxygen inhibited layer which leads to porosity and breakdown with time. On the other hand OS with double the amount of fillers claims that the primer can penetrate deeply into fissures of the teeth, resulting in a long lasting coverage and mechanical retention, in addition to its ability to release continuous fluoride to the tooth surface.[16] PS is 18% filled, and OS is 38% filled. It is well known that filler size and loading influence the wear properties of resin composite materials.[17]

According to SEM observation, the surfaces of OS and PS appeared very different in terms of particle size, shape and their organization within matrix [Figure 6]. OS composites showed a much higher particle volume fraction than PS composites reflecting the wear pattern observed with both profilometer and SEM. After wear, PS surface exhibited more flatness than OS [Figure 7]. Our wear resistance results showed significant differences between products, PS having significantly higher wear resistance ability compare to OS ($P < 0.01$) [Figure 3]. Results of the wear test and SEM images in this study are similar to that of microfilled and hybrid resin composite. The former contain submicron particles, while the latter contains the particles up to 4 μm.[18] As with microfilled resin composite, PS showed better wear resistance than

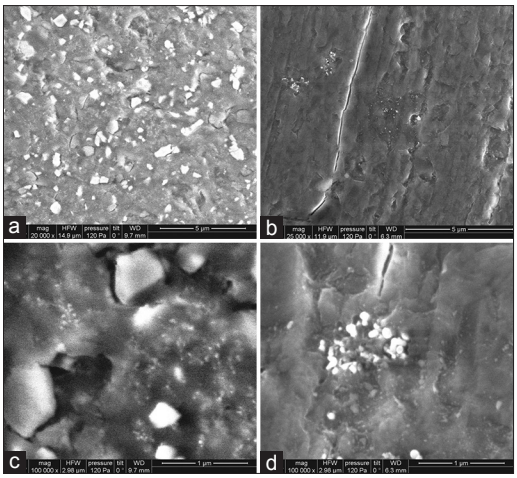

Figure 6: Scanning electron microscopy images for Opal® Seal (a) ×20,000, (c) ×100,000; L.E.D. Pro Seal (b) ×20,000, (d) ×100,000/away from the wear track (control surface)

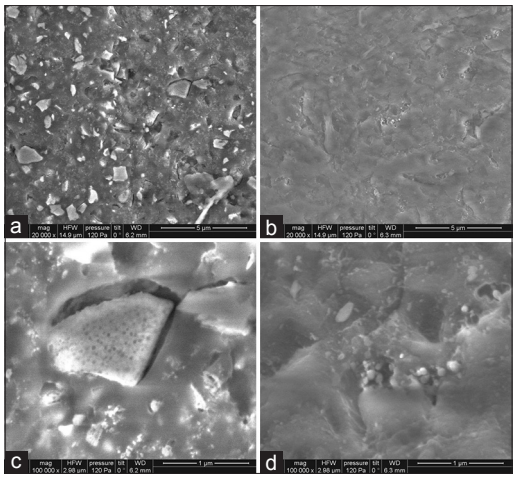

Figure 7: Scanning electron microscopy images for Opal® Seal (a) ×20,000, (c) ×100,000; L.E.D. Pro Seal (b) ×20,000, (d) ×100,000 on the areas of wear

OS since once these large particles of OS are plucked out, the volume loss of the surface is considered to be greater than the materials with smaller particle sizes such as microfilled or nanofilled resin composite, showing deeper wear patter in OS [Figure 7c]. However, the relationship between the amount of filler content and wear resistance properties was not linear.[19] This indicates there is a threshold limit for filler content to provide wear resistance while being hard. Bond strength between filler particles and resin matrix has a strong impact on wear.[20] Wear resistance properties are shown to be increased until the product reaches a saturation point, after which wear resistance properties decrease with increased filler content.[19] However, the important question is how much filler is considered to be desirable to have optimum wear resistance properties with required hardness.

In the present study, instead of a tooth brush, nylon rod was used to create a wear tract, and the volume of wear area was measured using a profilometer. This result was in agreement with another tooth brush wear resistance study, which compared PS and OS.[13] PS showed significantly less wear compared to OS in all time points tested from 3to 36 months.[13] Utilization of the contact profilometer for the wear test is considered to be less technique sensitive and cost effective, providing simple and clear determination of wear resistance of materials compared to the method of measuring the weight of samples before and after the wear or use of three-dimensional profilometer.

Our study indicated as expected that OS exhibited approximately one and half times more hardness with KHN [Figure 1] showing significance difference ($P < 0.001$) between products. This result is explained by OS's larger particle sizes and higher filler loading relative to PS. When performing hardness test, the diamond indent will have a higher probability of contacting the filler particles, rather than the matrix in OS samples. Since the hardness of particles is much greater than that of the matrix, OS exhibited a higher micro hardness values. In contrast, the probability of contacting the matrix during hardness testing is considered to be higher in PS with lower filler loading and smaller particle sizes.

With increased fillers, the color stability would be expected to be higher due to the lower percentage of the resin matrix. Both products show some color instability when stored in water. However, OS shows more stable results over time compared to PS, which showed a continuous increase in color within the experimental time duration. This difference may be explained by the adsorption property of the resin that was saturated faster with the pigments in OS due to the smaller amount of the matrix.[21] The measurable differences in color for OS were minimal and once stained, it remained the same over the period. The result of our color stability test relates to color properties of microfilled and hybrid resin composite. Hybrid resin composite showed better color stability than that of microfilled because of the greater filler loading.[18] However, it should be noted that filler loading is not the only factor for color stability.[22] Chemical composition such as activator, initiator, inhibitor, pigment and difference in scattering and absorption may have effects on color stability.[22]

Overall our results indicate 18% filler compared to 38% filler, imparts statistically different material properties to the sealant. For a sealant to be used on facial surfaces of teeth for the prevention of WSL in orthodontic patients, wear resistance and color stability are two important properties to be considered desirable. Increased filler content does not necessarily increase durability.[19] In our studies, 18% filler showed more resistant to wear which indicate that it may last longer on the surfaces resisting oral environmental stresses. Thirty-eight percent filler content contributed to better color stability. The question still needs to be explored is how much filler particle with what sizes can produce a color stable product with increased wear resistance desirable for long-term stability.

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